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ELECTRODEPOSITED, ELECTROLESS, AND ANODIZED COATINGS ON BERYLLIUM

Battelle Memorial Institute
Columbus, Ohio

September 1964

September 1, 1964
DMIC Memorandum 197

ELECTRODEPOSITED, ELECTROLESS, AND ANODIZED COATINGS ON BERYLLIUM

DEFENSE METALS INFORMATION CENTER
BATTELLE MEMORIAL INSTITUTE
COLUMBUS, OHIO 43201

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R&D		
(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)		
1. ORIGINATING ACTIVITY (Corporate author) Battelle Memorial Institute Defense Metals Information Center 505 King Avenue, Columbus, Ohio 43201	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b GROUP	
3 REPORT TITLE Electrodeposited, Electroless, and Anodized Coatings on Beryllium		
4 DESCRIPTIVE NOTES (Type of report and inclusive dates) DMIC Memorandum		
5. AUTHOR(S) (Last name, first name, initial) Beach, John G.		
6 REPORT DATE September 1, 1964	7a. TOTAL NO OF PAGES 5	7b NO. OF REFS 26
8a. CONTRACT OR GRANT NO AF 33(615)-1121 b. PROJECT NO 8975 c d	9a ORIGINATOR'S REPORT NUMBER(S) DMIC Memorandum 197 9b OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
10 AVAILABILITY/LIMITATION NOTICES Copies may be obtained from DMIC at no cost by Government agencies and by Government contractors, subcontractors, and their suppliers. Others may obtain copies from the Office of Technical Services, U. S. Department of Commerce, Washington, D. C. 20230		
11. SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY United States Air Force, Research and Technology Division, Wright-Patterson Air Force Base, Ohio 45433	

13 ABSTRACT

Because of expanding interest in the uses for beryllium, the Defense Metals Information Center has summarized information on surface finishing and coating of this metal. Its light weight and high modulus position beryllium as a favorable structural material for aerospace needs. The performance of beryllium is benefitted by preferred surface finishing and coatings. Available information is presented on electroless, electrodeposited and anodized coatings on beryllium. (Author)

DD FORM 1 JAN 64 1473

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Beryllium						
Coatings						
Electrodeposited coating						
Electroless coating						
Anodized coating						
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TABLE OF CONTENTS

ELECTRODEPOSITED, ELECTROLESS, AND ANODIZED COATINGS ON BERYLLIUM

John G. Beach*

SUMMARY

Coatings are applied to beryllium for four principal reasons:

(1) To resist corrosion and oxidation

(2) To facilitate joining

(3) To improve resistance to wear

(4) To increase thermal emissivity.

Nonmetallic coatings are produced by oxidation at high temperature or by electrochemical anodic treatment. Metallic coatings are produced by electrodeposition, electroless plating, flame spraying, and metallurgical cladding.

INTRODUCTION

This memorandum was prepared to bring together information made available to DMIC on the prefinishing and coating of beryllium by electrodeposition, electroless plating, and anodizing, used to provide both metallic and nonmetallic coatings.

Because of its light weight, high modulus of elasticity, and good thermal and nuclear properties, beryllium is of interest as a structural material in aerospace systems, as a component of gyroscopes, and as components in nuclear systems. To obtain efficient use of beryllium in these applications, however, coatings often are needed.

Beryllium metal forms a "protective" oxide film with a limiting thickness of about 100 A (10^{-6} cm) when exposed to air at room temperature for about 2 hours. Beryllium metal resists attack by dry air, nitrogen, hydrogen, and carbon dioxide at temperatures up to 1500 F. It is also resistant to pure water (free of halogen ions) and liquid metals (free of oxygen).

Beryllium, however, is corroded by water at high temperature, such as may be used in nuclear systems, and by oxygen in liquid metals such as sodium, which is used for heat transfer, unless the oxygen content is maintained at a very low level (<10 ppm). Beryllium is corroded by the fluorinated oils used in gyroscopes. It also is corroded by water containing as little as 0.5 ppm of chloride (considerably less chloride than that found in city or river water), and it pits in sea water. Beryllium has lower emissivity than is desired in space systems. The performance of beryllium in all of these environments can be improved by the application of coatings. For joining by soldering or by pressure bonding at moderately low temperatures, it is necessary to coat beryllium with a suitable metal. Increased wear resistance also can be obtained by coatings on beryllium.

There is, therefore, considerable need to develop coatings of various types for beryllium. Work no doubt will be continued on such problems, and additional information can be expected in the future.

Proper machining techniques[1] and safe handling procedures[2] should be observed with beryllium.

CLEANING AND SURFACE FINISHING

Unexplained corrosion of beryllium components can often be traced to bad practice in the manufacture of hardware.[3] Since the metal is extremely sensitive to corrosion by chlorides, surface cleanliness is extremely important. Detergent cleaning followed by distilled-water rinsing and drying can minimize corrosion of machine parts, which is attributed to a chloride-salt residue from tap water and/or "fingerprinting". Machined parts that were cleaned prior to exposure to one cycle of the MIL-E-5272 humidity test* showed little or no evidence of corrosion. Parts that were not cleaned showed considerable localized corrosion.[3]

Damaged metal in the surface layer of machined beryllium consists of microcracks and twinned areas. Although heat treatment can remove the effect of twinning damage on the mechanical properties of beryllium, it does not remove cracks, which will retain fluids and contaminants.

Mechanical lapping, if carried far enough, can overcome the microcrack problem. Smearing of the surface metal during machining or lapping must be avoided or contaminants will be entrapped. Diamond lapping compounds are recommended for beryllium. Generally, the removal of 2 mils of a machined surface is required to remove microcracks.

Chemical polishing of machined beryllium is effective in revealing and removing microcracks along with the twinned metal structure. However, chemical polishing will also selectively remove oxide particles and certain inclusions from the beryllium surface and thereby produce a pitted surface which at times can be less desirable than the microcracks and twins in the surface layers.

There is little doubt that, if maximum properties of beryllium parts are desired, cleaning and surface finishing should be included in the design specifications. Treatments that can be recommended for maximum properties include finishing cuts of 0.002 inch by machining followed by chemical etching or polishing to remove an additional 0.002 inch of the surface layer. Unless subsurface cracking is present, the above treatments can provide a crack-free beryllium surface.

Annealing at 1450 F and furnace cooling and/or chemical removal of the machine-damaged surface are effective methods to obtain maximum properties in beryllium.[4] However, in the case of highly wrought forms, such as extrusions or sheet, difficulties with stress relieving through twin formation are accentuated by annealing because of the formation of additional twins.[5]

The effects of surface condition on the hot-pressed properties of beryllium are illustrated by data on the following page.

Other data for beryllium that was originally sawed from a hot-pressed block show greater room-temperature tensile strength by more than 40 per cent, suggesting differences in material, the processing, and/or the testing procedures.[5]

* Senior Chemical Engineer, Electrochemical Engineering, Battelle Memorial Institute.

* Two hours' heating to temperature, 6 hours' exposure at 170 F to 100 per cent relative humidity, and 16 hours' cool-down and exposure to condensed moisture.

Room-Temperature Tensile Data[4]

Treatment of Beryllium (Y-5802)	UTS, ksi	YS, ksi	Contraction, %
I. Mechanical polish	31.5	25.4	0.2
II. I plus chemical polish	31.6	25.4	0.4
III. I plus vacuum anneal	31.5	26.4	2.0
IV. II plus vacuum anneal	31.7	25.8	1.7

1200 F Tensile Data[4]

Treatment of Beryllium (Y-6825)	UTS, ksi	YS, ksi	Contraction, %
V As-machined	16.6	14.9	2.9
VI. Chemical polish	16.9	15.8	7.6
VII. Vacuum anneal	20.1	17.7	15.0
VIII. VI plus VII	19.7	17.0	13.1

Room-Temperature Tensile-Impact Data[4]

Treatment of Beryllium (Y-4540)	Ft-lb at 11 ft/sec	Ft-lb at 17 ft/sec
Same as I above	1.3	0.2
Same as II above	4.5	3.2
Same as IV above	4.7	-

Reference 5

Treatment of Beryllium	UTS, ksi	YS, ksi	Elongation, % in 1 inch
V. As machined	45	36	1.0
IX. Ground (-2 mils)	51	39	1.3
X. Ground (-5 mils)	50	37	1.3
XI. V plus anneal	51	34	2.7
XII. IX plus anneal	54	36	3.0
XIII. X plus anneal	51	35	2.7
XIV. XI plus chemical etch	50	35	2.2
XV. XII plus chemical etch	53	35	3.2
XVI. XIII plus chemical etch	50	34	2.2

A beneficial effect of carefully removing the machine-damaged metal, 2 to 5 mils of the surface, is obvious from these data as a 50 per cent increase in the ultimate tensile strength. A beneficial effect of annealing to remove surface twins is also apparent, as an increase of about 100 per cent in ductility.

The effects of chemical surface finishing on the properties of beryllium and the performance of beryllium parts are not obvious, often because of the overriding effects that are attributed to the prior history of the part. In addition, the type of finishing solution and the operating conditions, along with the amount of metal removed, have been varied; thus, chemical-finishing effects are difficult to resolve with the available data.

COATINGS ON BERYLLIUM

Electrodeposited Coatings

Adherent electroplating on beryllium involves specific pretreatments of the metal surface.[4,6-15] Beryllium surfaces that are to be coated usually have been machined; therefore, gross scale and surface impurities are not a primary problem. If needed, the surface can be descaled by pickling in a hydrofluoric-nitric acid solution (2 vol % of 46% HF, 50 vol % of 70% HNO_3, and 48 vol % of water at room temperature).

Pretreatment of beryllium for adherent plating includes several precleaning and activation steps along with intermediate water rinses. Most metals that can be electrodeposited can be adherently plated directly on properly activated beryllium.[8] An alternative process involves depositing a chemical displacement zinc film (approximately 0.005 mil in thickness) on the beryllium surface prior to electroplating with copper and/or other metals.[8]

Since the majority of applications for beryllium have involved temperatures up to 1500 F, the metallurgy of the coated, composite system is important in the design of beryllium parts. Interdiffusion of a multi-metal system will result in continually changing interfacial layers. Thus, the ultimate operational characteristics of the composite system will be affected by the properties of the various alloys that are formed.

Nickel, iron, chromium, and silver offer promise as preferred metallic coatings on beryllium for elevated-temperature applications.[5,8] Nickel-coated beryllium shows no undesirable interfacial alloying after 30 days at 600 F. However, the diffusion between the nickel and beryllium that occurs in 30 days at 932 F results in brittle, low-strength, interfacial alloy layers.[8] Iron-coated beryllium shows alloying characteristics similar to that of nickel-coated beryllium but with slightly lower rates of diffusion.[8]

Chromium- or silver-coated beryllium, because of lower diffusion rates, offers good chance for electroplated metal coatings on beryllium to be used at high temperatures, above 900 F. However, electroplating techniques for applying these metals on beryllium for protection against oxidation at 1300-1400 F have not been developed yet. Four metallic and nonmetallic coatings which were appraised for the protection of beryllium at 1325 F in an argon-plus-air atmosphere (1 vol % air) gave the following life in hours:[16]

Diffused chromium (chromized) coating - OK at 405 hours, failed by oxidation by 580 hours

Chromized plus 8-mil electroplated nickel coating - nickel separated from chromized coating within 405 hours

AI* anodized coatings - failed within 405 hours

BBC** anodized coatings - OK at 840 hours

Several pretreatment cycles have been developed for adherent electroplating on beryllium. Procedures published by Kolodney in 1952,[6] by Beach and Faust in 1953,[8] and by Missel in 1960[11] constitute the major published plating technology for beryllium. Specific processing details[8] and modifications[4] follow.

Precleaning for Activation and Plating

Greases and oils are best removed by organic-solvent degreasing (vapor or contact). Residual

* Atomics International proprietary process.

** The Brush Beryllium Company proprietary process.

dirt is removed by cathodic cleaning in alkaline detergent solutions. Proprietary cleaning solutions containing caustic, carbonate, and/or wetting agent, such as those devised for aluminum, copper, magnesium, etc., can be used satisfactorily.

Zinc-Immersion Method[8]

After precleaning and water rinsing, the clean beryllium part is immersed in the following solution at 185 ± 5 F for about 5 minutes to produce the thin, uniform zinc displacement film on the surface:

Sodium tetropyrophosphate - $Na_4P_2O_7$ - 120 g/l
Zinc sulfate - $ZnSO_4 \cdot 7H_2O$ - 40 g/l
Sodium fluoride - NaF - 7.5 g/l
Potassium carbonate - K_2CO_3 - 5 g/l
pH 7.5-8.0 with sulfuric and/or phosphoric acid.

Other zinc solutions have been used with reported success.(10,12)

The adherent zinc film is a basis for subsequent electroplating with copper and/or other metals from solutions designed for plating on zinc. Examples of satisfactory plating baths and conditions for electroplating over zinc immersion-coated beryllium, after water rinsing, are:

Copper

Sodium cyanide - NaCN	46 g/l
Copper cyanide - CuCN	26 g/l
Potassium carbonate - K_2CO_3	15 g/l
Potassium hydroxide - KOH	7.5 g/l
Sodium fluoride - NaF	22.5 g/l
pH	13.2 ± 0.1
Temperature	130 F

Current density: 30 amp/sq ft for 1 min, followed by 15 amp/sq ft thereafter

Iron

Ferrous sulfate - $FeSO_4 \cdot 7H_2O$	300 g/l
Ferrous chloride - $FeCl_2 \cdot 4H_2O$	42 g/l
Ammonium sulfate - $(NH_4)_2SO_4$	15 g/l
Boric acid - H_3BO_3	35 g/l
Sodium formate - Na_2COOH	15 g/l
Duponol ME (Du Pont)	1 g/l
pH	4.0 ± 1
Temperature	140 F
Current density	40 amp/sq ft

Nickel

Nickel sulfate - $NiSO_4 \cdot 7H_2O$	143 g/l
Magnesium sulfate - $MgSO_4 \cdot 7H_2O$	75 g/l
Ammonium chloride - NH_4Cl	15 g/l
Boric acid - H_3BO_3	15 g/l
XXXD (Harshaw Chemical Co.)	20 ml/l
pH	5.5 ± 0.1
Temperature	90 F
Current density	15 amp/sq ft

Zinc

Zinc sulfate - $ZnSO_4 \cdot 7H_2O$	240 g/l
Ammonium chloride - NH_4Cl	15 g/l
Aluminum sulfate - $Al_2(SO_4)_3 \cdot H_2O$	30 g/l
Magnesium sulfate - $MgSO_4 \cdot 7H_2O$	70 g/l
Licorice	1 g/l
pH	4.0
Temperature	80 ± 5 F
Current density	25 amp/sq ft

Direct Plating Method I[8]

After being precleaned and water rinsed, the beryllium part is treated as follows:

(a) Anodic pickle
Phosphoric acid (85% H_3PO_4), 10% (by volume)
Hydrochloric acid (38% HCl), 2% (by volume)
Temperature, 80 ± 10 F
Current density, 15 ± 5 amp/sq ft
Time, 1 min

(b) Chemical pickle (without rinsing)
Concentrated nitric acid (70% HNO_3)
Temperature, 80 ± 10 F
Time, 2 min

(c) Water rinse

(d) Acid dip
Ammonium sulfate - $NH_4(SO_4)_2$, 100 g/l
Sulfuric acid - H_2SO_4, 10 g/l
Temperature, 80 ± 10 F
Time, 1/2 to 1 min

(e) Water rinse

(f) Electroplate

Examples of satisfactory baths and conditions for electroplating directly on activated beryllium are:

Nickel and Iron

(See previous section)

Aluminum

Aluminum can be plated directly on pretreated beryllium from nonaqueous organic electrolytes.

Chromium

Chromic acid solutions passivate beryllium surfaces. Therefore, chromium is plated over another intermediate metal such as copper on beryllium.

Silver Strike Bath

Silver cyanide - AgCN	4.5 g/l
Sodium cyanide - NaCN	70 g/l
Temperature	80 ± 10 F
Current density	7.5 amp/sq ft
Time	5 minutes

Silver Plating

Silver cyanide - AgCN	75 g/l
Potassium cyanide - KCN	112 g/l
Potassium carbonate - K_2CO_3	22.5 g/l
pH	13.0 with KOH
Temperature	120 F
Current density	25 amp/sq ft

Tin

Sodium stannate - $Na_2SnO_3 \cdot 3H_2O$	150 g/l
Sodium hydroxide - NaOH	15 g/l
Sodium acetate - $NaC_2H_3O_2$	22.5 g/l
Temperature	1[illegible] F
Current density	25 amp/sq ft

Copper

Sodium cyanide - NaCN	30 g/l
Copper cyanide - CuCN	22.5 g/l
Sodium carbonate - Na_2CO_3	15 g/l
Sodium sulfite - $Na_2S_2O_3$	0.5 g/l
Temperature	120 F
pH	9 with tartaric acid
Current density	25 amp/sq ft

Direct Plating Method II[11]

Nickel coatings (7 mils in thickness) on beryllium have withstood a solar-furnace test in which the underlying beryllium could be heated to its melting point (2341 F) in about 9 seconds. The following processing sequence was reported for adherent nickel plating on beryllium:[11]

(a) Abrasive clean
   Wet 400-grit emory paper

(b) Water rinse

(c) Anodic clean
   5 minutes with current density of 50 amp/sq ft in a mild brass-type cleaner at 130 F

(d) Water rinse

(e) Acid etch
   Nitric acid (70% HNO_3) - 5 vol %;
   Hydrofluoric acid (48% HF) - 1 vol %;
   Water - 94 vol % at room temperature
   30-second immersion

(f) Water rinse

(g) Acid activate
   Sulfuric acid - 3.6N H_2SO_4 at room temperature;
   30- to 60-second immersion

(h) Water rinse

(i) Nickel plate
   Proprietary all-sulfamate bath
   pH 3.0 to 3.5;
   Temperature, 130 F;
   Current density, 50 amp/sq ft

(j) Water rinse and dry

Electroless Coatings

Electroless Nickel

Electroless nickel on beryllium is enjoying considerable popularity as an extremely hard and wear-resistant coating.[4,13] The coating, a nickel-phosphorus alloy containing 6 to 8 per cent phosphorus in solid solution, is nonmagnetic until heated to 750 F and above. The alloy has a coefficient of thermal expansion of 7.2×10^{-6} in./in./F, which compares favorably with that of beryllium, 6.3×10^{-6} in./in./F.

Camera mirrors are electroless-nickel coated, polished, and flash-aluminum coated for aerospace needs. The "Kanigen" electroless-nickel coating polishes like glass, and the thin, vacuum-deposited aluminum provides the needed tarnish resistance.[17] Gyro components were one of the earliest uses of electroless-nickel-coated beryllium. The coating aided wetting of the beryllium component by "potting" compounds.[17]

Pretreatment of beryllium for electroless-nickel plating involves precleaning and application of an immersion zinc film.[18] Kanigen nickel plating is accomplished without any intermediate coating.[17] Reported data on electroless-nickel-coated beryllium are very limited.

Electroless Platinum

Platinum-black coatings on beryllium were investigated by Missel and Greear to consistently provide a reliable high, total, infrared emittance surface (>0.8) for large parts.[19] The following processing steps were developed and adapted to the coating of hemispherical parts of beryllium using spray-coating techniques:

(a) Precleaning
   1. Abrade with wet emory paper or cloth of 180 mesh or finer
   2. Alkaline clean and water rinse

(b) Activation
   1. Acid treat with 3.6N H_2SO_4 for 2 to 3 minutes and water rinse
   2. Zincate treat with zinc chloride solution for 1/2 to 1 minute and water rinse

(c) Black platinizing
   1. Treat with chloroplatinic acid solution for about 2 minutes and water rinse
   2. Alcohol rinse and dry.

All solutions are at ambient temperature. Drying after Steps (a2), (b1), or (b2) apparently is not detrimental. The zincate solution (b2) contains 100 g/l of $ZnCl_2$ in water adjusted to a pH of 4.9 ± 0.1 with acetic acid. The platinizing solution contains 10 g/l of chloroplatinic acid in water.

Anodized Coatings

Oxide coatings have been studied rather extensively for protecting beryllium and for providing control of the thermal-radiation properties for use in spacecraft.[16,20-26]

Air oxidation of beryllium at 900 to 1500 F produces normal hexagonal BeO platelets, parallel to the (0.001) plane, randomly disposed and several hundred angstroms in diameter.[20] Such oxide coatings are not protective. Anodically produced oxide coatings (nitric-chromic acid solutions), also BeO, increased in thickness linearly with the applied voltage.[20] The coating as deposited is not a dielectric. Later studies showed that the anodized layers are crystalline and grow as platelets with a mean diameter of ~60 A and a mean thickness of ~20 A.[21]

Chromic acid anodizing of beryllium produces an adherent, glossy-black film of BeO about 0.1 mil in thickness.[22,23] Such coatings were proposed for corrosion protection, for a paint base, and for a heat-radiative surface.[23]

Anodized beryllium was investigated to prevent or retard the interaction of beryllium with uranium dioxide and corrosion by moist CO_2 at temperatures above 1200 F.[24] Better results were observed with chromic acid anodizing than with nitric-chromic acid anodizing. Reaction with UO_2 was avoided. Corrosion in moist CO_2 (3-4 vol % H_2O) at 1200 F was reduced, but not consistently.

The reflectance of anodized beryllium (1% Cu) is relatively low at the short wave lengths and high at the longer wave lengths; thus, anodized beryllium is attractive for use as a solar heat-collector surface.[25] Sodium hydroxide anodizing in these studies showed more promise than chromic acid anodizing of the beryllium-1% copper alloy.

A proprietary anodizing process for beryllium has been developed and has been shown superior to other anodizing processes for certain needs.[26] This BBC anodizing process was demonstrated capable

for adequate coating and for protecting the intricate neutron-reflector shapes of beryllium that are of interest for the SNAP-8 program.

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